

## Preparative Purification of Flavanone by Semi-preparative Unified Fluid Chromatography System and Cyclone Separator-87 (CS-87)

### Introduction

Preparative purification in SFC has advantages for high throughput measurements and post-collection fraction treatment. In terms of post-collection fraction treatment, SFC uses CO<sub>2</sub> as mobile phase, so evaporation to dryness can be easily carried out by drying off the co-solvent (typically an alcohol). CO<sub>2</sub> has a low-cost and high purity, which allows measurement with less impurities and reduces solvent costs.

CO<sub>2</sub> has a 500 times adiabatically-expansion when released into atmospheric pressure at exit of pressure adjustment valve, thus it is important to properly control this expansion to maximize sample recovery. Cyclone Separator-87 (CS-87), gas-liquid separator, provides a high recovery rate for analysis at less than 50 mL/min CO<sub>2</sub> flow rates.

In this application, chiral preparative purification of flavanone using the Prep SFC System-M, CS-87, and fraction collector management program for EXTREMA UFC is reported.

**Keyword:** Flavanone, Prep-SFC, UFC, CS-87, ChromNAV Ver.2, Fraction collector management program, Chiral separation

### Experimental Condition

Column: CHIRALPAK IA  
(20 mmI.D. x 250 mmL, 5 μm)

Eluent: CO<sub>2</sub>: 50 mL/min, methanol: 10 mL/min

Column temp.: 40°C

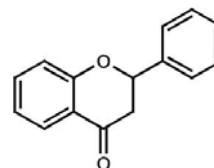
Wave length: UV: 210 nm

Back pressure.: 10 MPa

Injection volume: 1000 μL

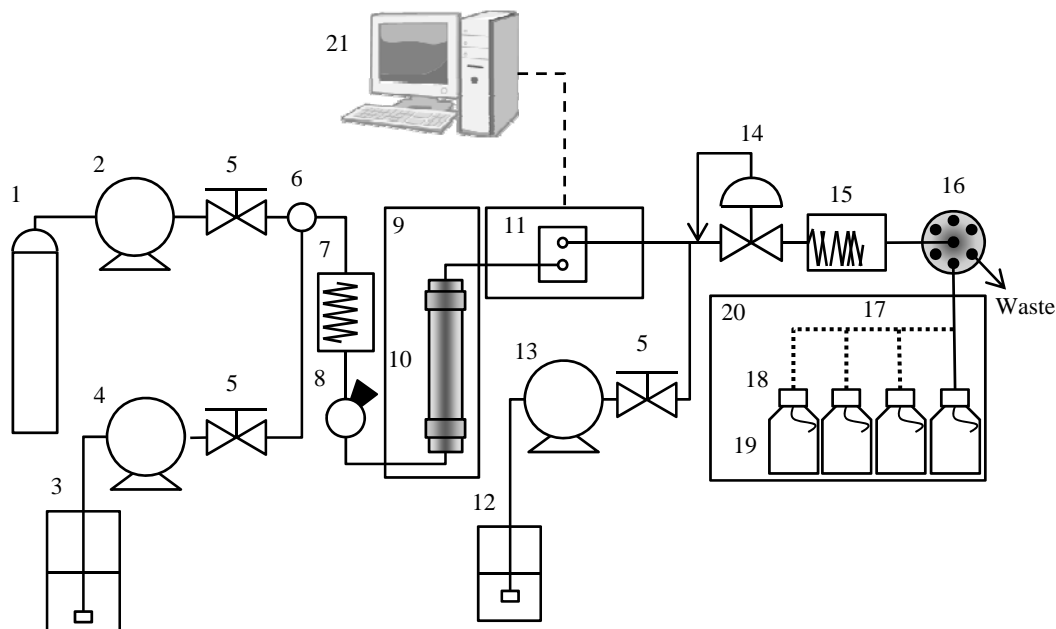
Standard: 1 mg/mL flavanone in methanol

### Structure



Flavanone

## Schematic diagram



- 1: CO<sub>2</sub> cylinder, 2: CO<sub>2</sub> pump, 3: Modifier solvent, 4: Modifier pump, 5: Stop valve, 6: Mixer,  
 7: Pre-heater (HE-01), 8: Autosampler, 9: Column oven, 10: Column, 11: UV detector, 12: Make up solvent,  
 13: Make up pump, 14: Back pressure regulator, 15: Post-heater (HE-02), 16: Switching valve,  
 17: Fraction collector, 18: Cyclone separator-87 (CS-87), 19: Duran bottles, 20: Fume Hood,  
 21: Chromatography data system (ChromNAV Ver.2)

Figure 1 shows the Cyclone Separator-87 (CS-87), gas-liquid separator. CS-87 provides easy installation, repeatedly use and significant increase in fraction recovery.



Fig. 1 Cyclone Separator-87(CS-87)

## Result

Figure 2 shows the chromatogram of flavanone recovery, and figure 3 shows the re-chromatogram of recovery liquid. As shown in figure 3, contamination is not observed, showing that the CS-87 provides high performance with high recovery.

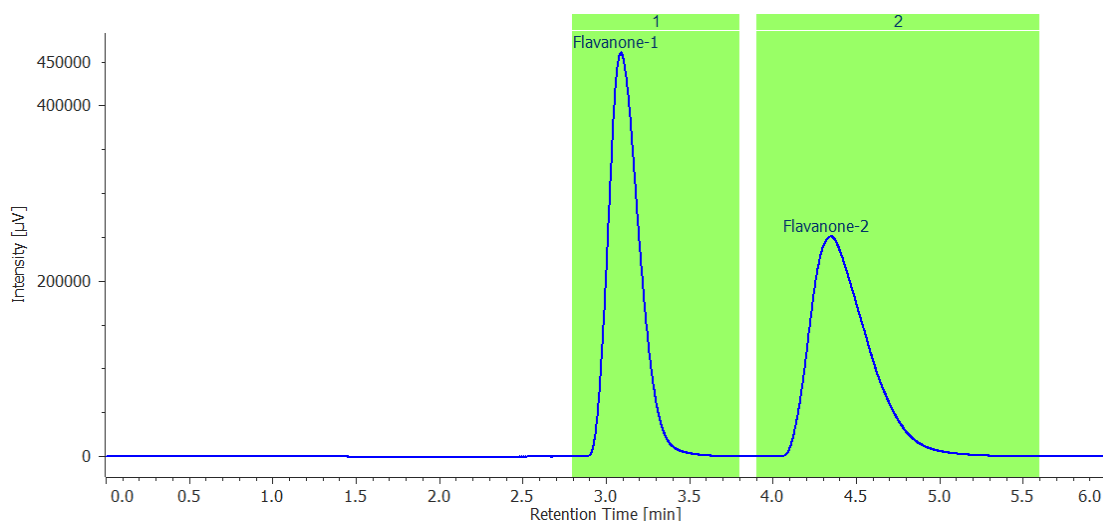


Fig. 2 Fraction of flavanone

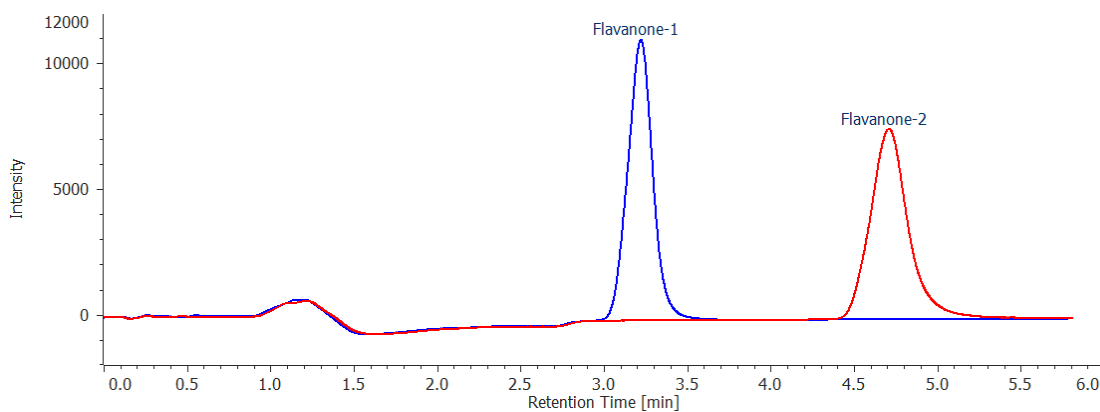


Fig. 3 Re-chromatogram of flavanone recovery liquid

Table 1 shows recovery rate of flavanone. As shown in the table Flavanone-1 and 2 shows high recovery rate.

	Flavanone-1	Flavanone-2
Recovery rate(%)	99.6	99.8

Table 1 Recovery rate of flavanone